APPLICATION NOTE 65906

Automated micro-SPE clean-up for GC-MS/MS analysis of pesticide residues in cereals

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Keywords: Pesticide residues, cereals, QuEChERS, micro-solid phase extraction (µSPE), GC-MS/MS, advanced electron ionization (AEI), targeted quantitation, TSQ 9000, Chromeleon Chromatography Data System

Goal

To assess the suitability of an automated micro-solid phase extraction (µSPE) clean-up of QuEChERS extracts for the determination of pesticide residues in cereal samples by gas chromatography coupled to triple quadrupole mass spectrometry.

Introduction

Worldwide food demand is set to increase substantially in the next few decades[1], and consequently, food safety concerns are also growing quickly[2,3]. To meet the demand for food, pesticides are used to control pests and ensure high crop yields, but there are some concerns that banned pesticides are still used illegally. If used incorrectly, pesticides can affect consumer's health, hence the importance regulatory bodies place on screening food samples for the presence of pesticide residues.

Given the large number and types of food samples that need to be tested, any delays in the analysis could ultimately impact the timely import/export of food products, which is crucial for perishable products. The extraction of pesticides from food matrices is typically carried out using the QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) acetonitrile method. Many versions of QuEChERS have been published but one of the most widely used versions is AOAC 2007.01[4]. This method includes a manual dispersive solid-phase extraction clean-up step (dSPE) of the initial non-cleaned extract. This clean-up procedure can be time-consuming and can result in limited removal of matrix co-extractives. By replacing this manual clean-up step with an automated µSPE clean-up approach, laboratories can save time, achieve more effective removal of co-extractives, and thus improve the consistency of the results. A miniaturized SPE method, consisting of sorbents

contained in a miniaturized cartridge, was first introduced by Morris and Schriner[5] for LC-MSMS analysis. Lehotay *et al*[6] and Goon *et al*[7] have since published workflows based on the use of miniaturized SPE clean-up of QuEChERS extracts before GC-MS/MS analysis.

This work was aimed at assessing the suitability of an automated µSPE clean-up approach of QuEChERS extracts of rice and wheat samples for the multi-class determination of a large number of pesticides. The cleaned-up extracts were analyzed using a Thermo Scientific™ TSQ™ 9000 triple quadrupole GC-MS/MS system equipped with the advanced electron ionization source (AEI). The sample introduction and automated clean-up were performed using a Thermo Scientific™ TriPlus™ RSH robotic autosampler configured with a liquid injection tool as well as with the dedicated µSPE tool and cartridges tray for automated clean-up. Data acquisition and processing were carried out using the Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS) software, version 7.2.

Experimental

GC-MS/MS analysis

A gas chromatograph (Thermo Scientific™ TRACE™ 1310 GC) was coupled to a TSQ 9000 triple quadrupole GC-MS/MS. The GC conditions are given in Table 1a, the MS parameters are detailed in Table 1b, and the autosampler parameters in Table 1c.

Glassware, reagents, and chemicals

- Anhydrous $MgSO_4$, Thermo Scientific™ (P/N 80020-415-500)
- Acetonitrile, Optima™ LC/MS Grade, Fisher Scientific™ (P/N A955-4)
- Acetic acid glacial (certified ACS), Fisher Scientific™ (P/N A38S-500)
- QuEChERS Salts (2007.01) mylar pouch 6 g magnesium sulfate (anhydrous),1.5 g sodium acetate, Thermo Scientific™ HyperSep™ (P/N 60105-341)
- µSPE GC cartridges 45 mg: 20 mg MgSO4, 12 mg PSA, 12 mg C18 and 1 mg CarbonX (P/N 60101-45GC)
- 2 mL screw vial kit, clear glass vials with caps, Thermo Scientific™ (P/N 60180-599)
- Screw caps with PTFE starburst slitted septum (LEAP PAL Parts + Consumables™, CAP-ND9-ST-SP10SB-100)
- Mixer grinder (Maharaja™ Whiteline, Delhi, India)

Table 1a. GC instrument conditions[8]

TRACE 1310 gas chromatograph parameters	
Column	Thermo Scientific™ TraceGOLD™ TG-5SILMS with 5 m SafeGuard, 30 m × 0.25 mm ID × 0.25 µm (P/N 26096-1425)
Injector	Split/Splitless (SSL)
Liner	SSL Splitless liner, single taper (P/N 453A1925UI)
Injector mode	Splitless with surge
Splitless time	0.3 min
Surge pressure and time	250 kPa for 1 min
Injection volume	1.0 µL
Injector temperature	250 °C
Column flow	1.20 mL/min
Carrier gas and purity	Helium (99.999%)
Purge flow	5.00 mL/min
Split flow	50.00 mL/min; Gas Saver Flow 10 mL/min after 10 min
Total run time	33.4 min
GC oven program	90 °C, 3 min 25 °C/min to 180 °C 5 °C/min to 280 °C 10 °C/min to 300 °C, 5 min

Table 1b. Mass spectrometer parameters

TSQ 9000 mass spectrometer parameters	
Acquisition mode	Timed selected reaction monitoring (t-SRM)
MS transfer line temp.	300 °C
Ion source temp.	320 °C
Ion source	AEI (Advanced Electron Ionization)
Electron energy	70 eV
Ionization	Electron Ionization (EI)
Collision gas and pressure	Argon at 70 psi
Peak width	0.7 Da (both Q1 and Q3)
Tune	AEI SmartTune

Table 1c. Autosampler parameters[6]

TriPlus RSH autosampler parameters	
µSPE sample load volume	300 µL
µSPE sample fill speed	20 µL/s
µSPE sample load speed	2 µL/s
µSPE sample vial penetration depth	30 mm
Mixing cycles	5
Mixing speed	20 µL/s
Mixing volume	250 µL
Pre-wash solvent	Acetonitrile
Pre-washing cycles	2
Post-wash solvent	Acetonitrile:Methanol:Water (1:1:1)
Post-wash cycles	5
Injection mode	Air plug

Sample preparation

Rice and wheat were purchased locally. Both types of samples were ground and homogenized separately to achieve a consistent particle size of approximately 200–500 µm. Rice has a high content of carbohydrate (80%), protein (7%), fat (2%), and fiber (11%); whereas, wheat has fewer carbohydrates (71%), more protein (12.6%), and similar fat (1.5%) and fiber (12%) amounts. Sub-samples (5 g) of the homogenized sample were weighed into a centrifuge tube and then spiked with pesticides at the concentration of 0.01 mg/kg. Water (10 mL) was added to rehydrate the sample to ensure the moisture content is enough for effective liquid-liquid partitioning on the addition of 1% acetic acid in acetonitrile (15 mL). After extraction and centrifugation, the extract supernatant was frozen at -20 °C to freeze out lipid co-extractives. The samples were centrifuged at -5 °C and an aliquot of supernatant cleaned up using µSPE, as outlined in Figure 1.

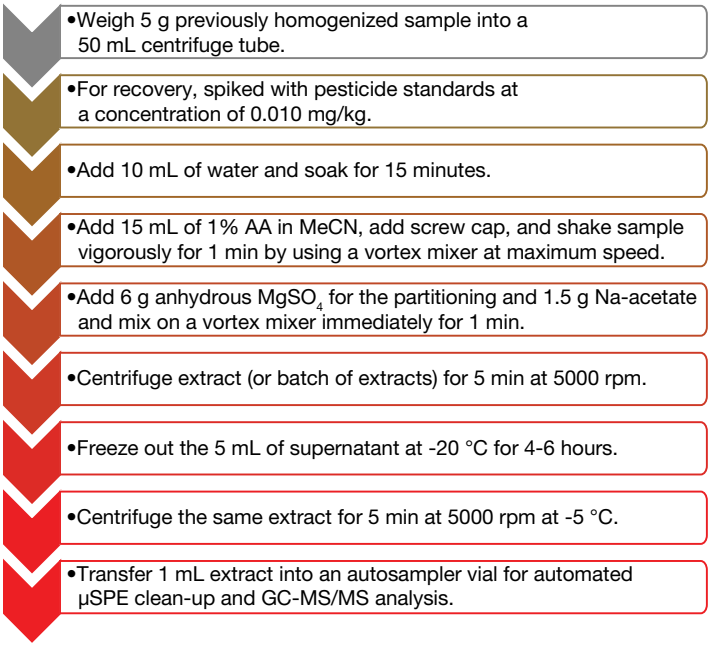

Figure 1. Sample preparation workflow before automated µSPE clean-up

Automated µSPE clean-up

Automated µSPE was performed using a TriPlus RSH autosampler fitted with the Thermo Scientific µSPE GC QuEChERS clean-up kit (P/N 1R77010-1160), including 1 mL volume syringe for solvent/sample dispensing and dedicated aluminum trays for cartridges and clean extract collection. The µSPE cartridge (P/N 60101-45GC) containing a total of 45 mg of an optimized sorbent blend (20 mg $MgSO_4$, 12 mg PSA, 12 mg C18, and 1 mg CarbonX) were used for clean-up. The µSPE clean-up reduces the number of steps and requirement for manual input as shown in Figure 2. The µSPE cartridges and the sample extracts (2 mL in each glass vial) were placed in the allocated positions on the corresponding TriPlus RSH autosampler trays. A volume of 300 µL of the sample extract was aspirated by the syringe first, and then the cartridge was transferred to the dedicated tray, where the cartridge was inserted into 2 mL glass vials with pre-split septa. The sample was loaded onto the cartridge for the clean-up. The sample extract was collected in a collecting vial and mixed with five cycles of mixing (pumping) with a 1 mL syringe. Then using a 10 µL syringe, 1 µL of the cleaned-up extract was injected into the GC-MS/MS. One advantage of µSPE vs manual SPE is that the solvent evaporation step is not needed. Further details are given in Table 2 and Figure 3. Preconditioning, loading, transfer, and elution of µSPE cartridges are performed automatically by the robotic autosampler during the analysis of the previous sample, with no increase in analysis cycle time (Figure 2).

Preparation of calibration standards

- Solvent standard calibration: The solvent standard calibration was prepared at 0.0025, 0.005, 0.01, 0.025, 0.05, and 0.1 mg/L.
- Matrix-matched calibration standards (µSPE): Aliquots of blank matrix extract were spiked after the initial extraction and before clean-up. The matrix-matched calibration was prepared at 0.0025, 0.005, 0.01, 0.025, 0.05, and 0.1 mg/kg as per the procedure given in the Thermo Scientific Application Note[9]. The non-cleaned matrix-matched calibration solutions were placed in vials and then loaded onto the autosampler tray for clean-up and GC-MS analysis.
- Sample extracts, as well as a matrix-matched standards, blank, and recovery spiked extracts were analyzed by GC-MS/MS.

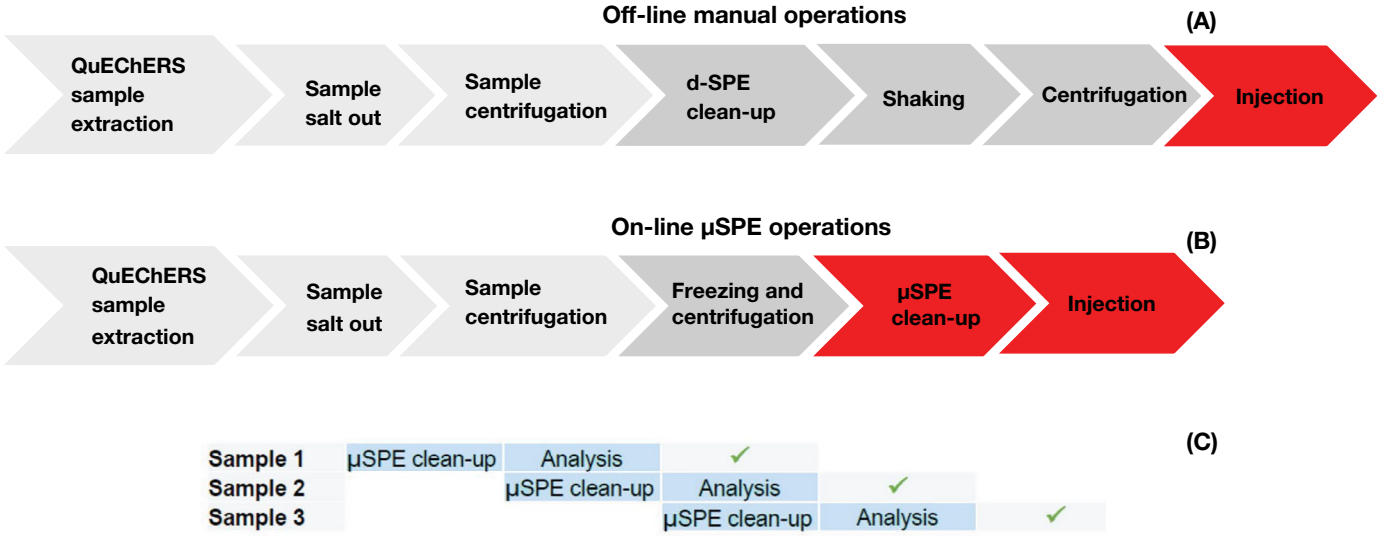

Figure 2. Sample extraction procedure as per the AOAC 2007.01; A) Manual clean-up with dSPE; B) Automated sample clean-up with µSPE; C) Script for the sample overlaid analysis and µSPE clean-up

Table 2. TriPlus RSH autosampler cycle used for automated µSPE clean-up

No.	Step description	Time (s)
1	Parking of 10 µL syringe to the home position	40
2	Pick up of 1 mL syringe and move to the home position	20
3	Fast wash the 1 mL syringe with MeCN (2 pumps of 1 mL each)	60
4	Mixing the extract with 1 mL syringe (2 pumps of 0.25 mL each)	30
5	Load 300 µL extract from vial in Tray 1 into 1 mL syringe	40
6	Place mini-cartridge above collection vial (with glass insert) in Tray 2	10
7	Elute extract through mini-cartridge at 2 µL s^{-1}	150
8	Discard mini-cartridge into a waste receptacle	10
9	Mix the eluate with 1 mL syringe (5 pumps of 250 µL each)	100
10	Wash the 1 mL syringe with 1/1/1 MeCN/MeOH/water (2 pumps of 0.5 mL each)	30
11	Wash the 1 mL syringe with MeCN (4 pumps of 0.5 mL each)	45
12	Switch to 10 µL syringe and wash with MeCN (2 pumps of 5 µL each)	80
13	Wash the 10 µL syringe with 1/1/1 MeCN/MeOH/water (5 pumps of 5 µL each)	40
14	Wash the 10 µL syringe with extract (3 pumps of 3 µL each)	30
15	Mixing the extract with 10 µL syringe (2 pumps of 3 µL each)	15
16	Injection of 1 µL of cleaned extract to GC-MS/MS	10

Data acquisition and processing

The data acquisition and processing were carried out using the Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS) software, which allows instrument control, method development, quantitative analysis, and customizable reporting all within one package. The target list of analytes with their selected reaction monitoring (SRM) parameters is given in Appendix 2. The data were acquired in t-SRM mode, which includes a minimum of two or more transitions per analyte. For data processing, the ion ratio (± 30%), retention time (± 0.1 min), linearity (R^2 > 0.995 with residuals < ± 20%), recovery (70–120%) and precision (± 20%) were set as user-defined criteria as per the SANTE guidelines[10].

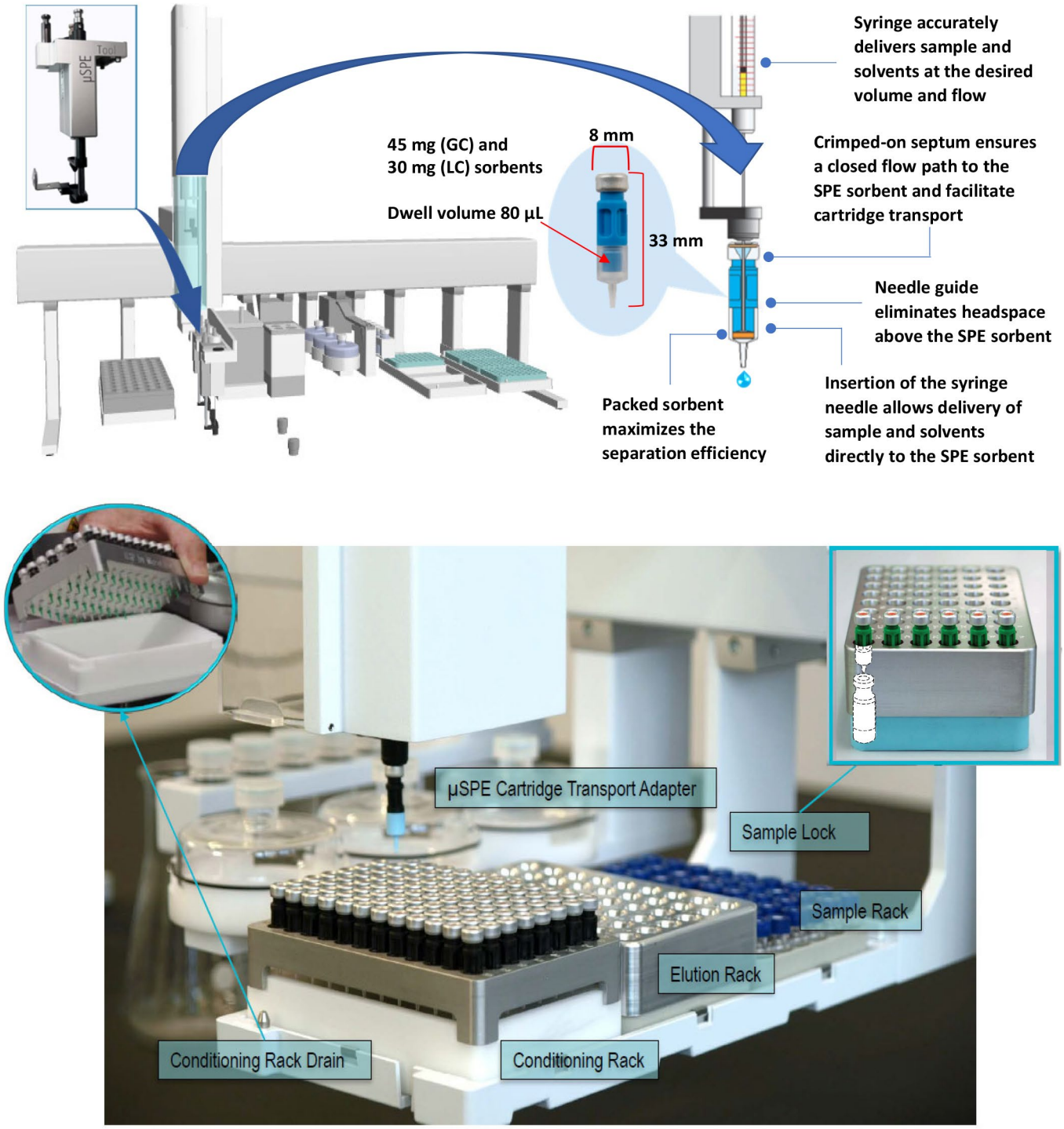

Figure 3. TriPlus RSH autosampler, µSPE tool operation with cartridge, and µSPE clean-up tray module

Results and discussion

Since the matrix-matched standards were subjected to the µSPE clean-up, the results are effectively corrected for any analyte losses on the cartridge but not losses during extraction. Nevertheless, this calibration approach improves accuracy and precision and is permitted by the EU SANTE guidelines. For identification, two SRM transitions per analyte were considered for all the target analytes at 0.01 mg/kg in rice and wheat with the retention time stability (± 0.1 min) and ion ratios (± 30%). The ion ratio (%) for ethalfluralin is represented in Figure 4.

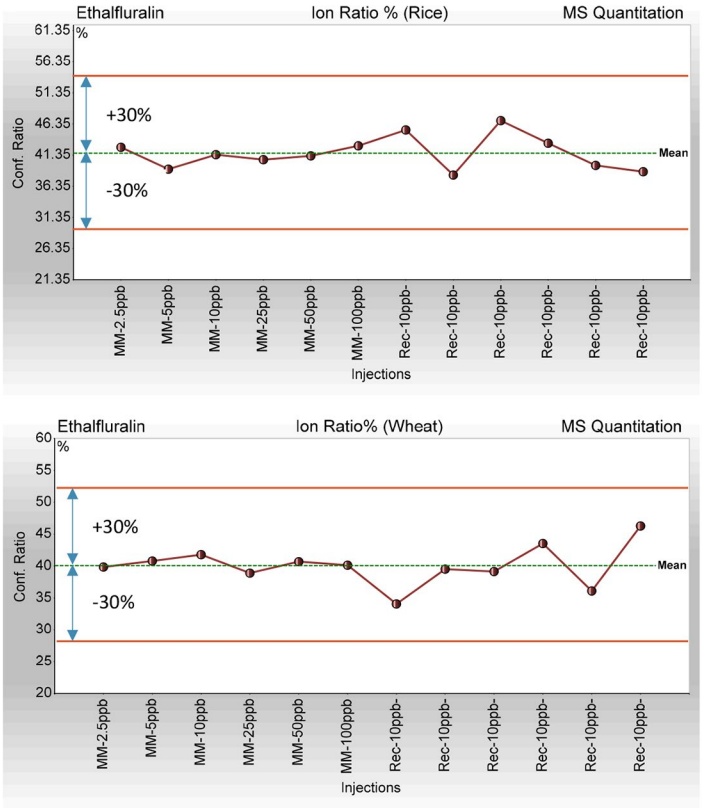

Figure 4. Ion ratio % for ethalfluralin in rice (top) and wheat (bottom) matrix-matched standards and recovery samples (n=6) pre-spiked at 0.01 mg/kg and subjected to µSPE clean-up. The EU SANTE ion ratio tolerance (%) is represented by red lines.

For confident quantification, maintaining peak symmetry with enough data points per peak is critical to achieving satisfactory repeatability. For example, the matrix-matched calibration standard of propachlor is shown in Figure 5 at 0.025 mg/kg. Also, propachlor provided excellent recovery and repeatability (RSD ≤ 4%, n=6) at 0.01 mg/kg (Appendix 1).

The matrix-matched calibration standards (rice and wheat) were linear over the concentration range of 0.0025 to 0.1 mg/kg. The coefficient of determination R^2 were mostly >0.995 with residual values (as % average calibration factors) of <20% for all target analytes. An example of linearity is shown for propachlor in rice and wheat (Figure 6).

The recoveries at 0.01 mg/kg in rice were in the range of 78 to 119% (n=6) with less than 20% RSD for all target analytes (Figure 7), except chlorothalonil and tolylfluanid, which gave low responses. These pesticides are known to be susceptible to instability during analysis. At 0.01 mg/kg, the recoveries (n=6) of pesticides in wheat were between 75 and 104%, with associated %RSD < 13% for 203 of 209 pesticides as shown in Figure 8.

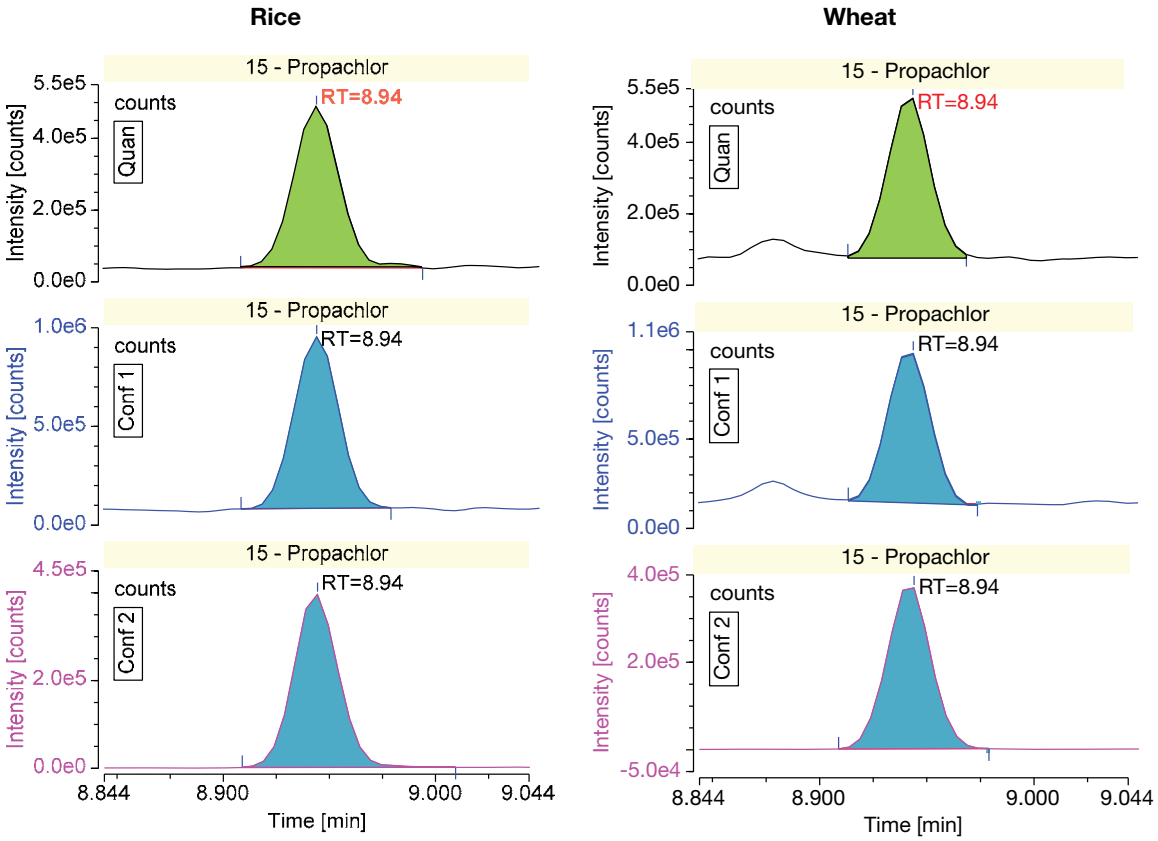

Figure 5. Response observed for propachlor (quantitative and confirmatory ions) in rice (left) and wheat (right) matrix-matched standards prepared with the µSPE clean-up at a concentration of 0.025 mg/kg

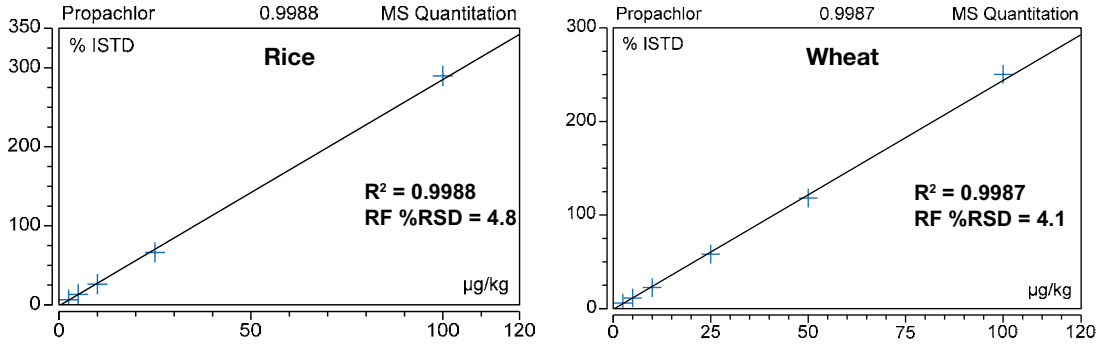

Figure 6. Calibration curve for propachlor analysis in rice and wheat matrix-matched standards with the µSPE clean-up over a concentration range of 0.0025 to 0.1 mg/kg

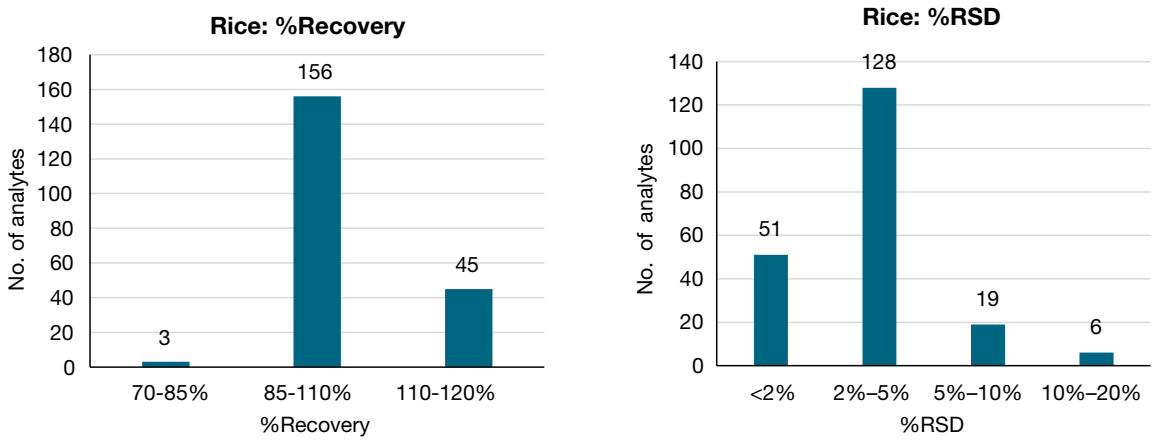

Figure 7. Recovery (%) and RSD (%) for rice matrix at 0.01 mg/kg followed by the µSPE clean-up

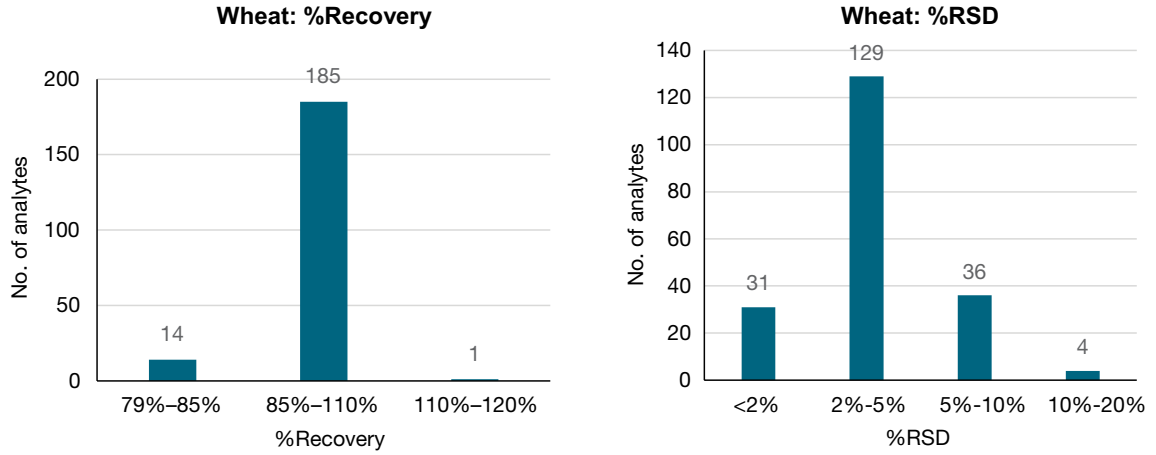

Figure 8. Recovery (%) and RSD (%) for wheat matrix pre-spiked at 0.01 mg/kg followed by the clean-up with µSPE

The vast majority of %RSDs at 0.01 mg/kg in rice and wheat were <5%. Information for individual pesticide-matrix combinations is given in Appendix 1. Overall, excellent recovery and precision values were obtained, which confirmed that the µSPE can be used as a replacement for the labor-intensive, more time-consuming dSPE manual clean-up method.

Conclusion

The experiments demonstrate that automated µSPE compared to dSPE with weighing the sorbents can significantly reduce the sample preparation time by 40 to 50% and increase sample throughput in routine laboratories by more than 1.5 times considering a batch of 10 samples. The miniaturized SPE cartridge features an optimized sorbent amount and composition, which acts with the optimum and controlled elution rate to provide high selectivity and high clean-up efficiency.

- Replacing the manual d-SPE procedures with µSPE delivers optimum recovery and precision while reducing the risk of human errors.
- The automated on-line clean-up workflow allows labor and time savings during sample preparation and increases unattended sample throughput in the laboratory.
- One cartridge type removes pigments, lipids, etc., so it is suitable for a large number of different sample types. Since it is not necessary to match the sample type to a specific blend of sorbents, the laboratory workflow is simplified.
- Excellent linearity was obtained using matrix-matched calibration standards over a concentration range of 0.0025 to 0.1 mg/kg with R^2 values mostly >0.995 and %RSD of residuals <5%.
- The performance has been checked with six replicates of pre-spiked samples at 0.01 mg/kg. The results (%recovery and %RSD) were in the range of 70 to 120% and <20%, respectively, and thus in compliance with the EU SANTE guideline criteria.

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Appendix 1. List of pesticides analyzed in rice and wheat results (retention time, linearity, recovery, and precision at 0.01 mg/kg) with the µSPE clean-up

			Rice (0.01 mg/kg)			Wheat (0.01 mg/kg)		
Sr. No.	Compound Name	RT	R^2	%Rec.	%RSD	R^2	%Rec.	%RSD
1	2,3,5,6-Tetrachloroaniline	9.19	0.9983	97	3.1	0.9980	92	2.9
2	2,4'-Methoxychlor	18.91	0.9960	108	1.3	0.9989	94	1.2
3	3,4-Dichloroaniline	7.55	0.9966	97	2.1	0.9988	79	2.9
4	4,4'-Methoxychlor olefin	18.32	0.9983	94	2.5	0.9987	94	1.4
5	Acetochlor	11.86	0.9940	111	6.7	0.9984	87	5.9
6	Acrinathrin	22.59	0.9977	105	1.9	0.9967	104	4.6
7	Alachlor	12.09	0.9955	111	2.7	0.9987	93	2.7
8	Aldrin	13.32	0.9993	87	2.2	0.9979	91	4.9
9	Allidochlor	6.47	0.9968	106	2.0	0.9990	96	2.3
10	Anthraquinone	13.32	0.9980	101	5.2	0.9941	89	3.2
11	Atrazine	10.31	0.9966	108	6.3	0.9990	91	4.6
12	Azinphos-ethyl	22.67	0.9988	113	2.8	0.9975	93	2.3
13	Azinphos-methyl	21.53	0.9956	115	2.3	0.9947	92	3.7
14	Benfluralin	9.21	0.9949	113	2.7	0.9963	91	2.8
15	BHC, Alpha	9.91	0.9973	101	1.0	0.9979	92	1.7
16	BHC, Beta	10.37	0.9971	99	2.6	0.9983	93	5.6
17	BHC, delta	11.16	0.9979	100	1.2	0.9981	93	3.2
18	BHC, gamma	10.60	0.9979	100	2.3	0.9963	90	3.4
19	Bifenthrin	20.36	0.9992	100	1.2	0.9984	96	2.2
20	Bromfenvinphos	15.58	0.9972	111	2.5	0.9988	99	4.6
21	Bromfenvinphos-methyl	14.38	0.9955	104	3.8	0.9987	100	5.5
22	Bromophos-ethyl	14.98	0.9978	107	3.0	0.9984	92	2.0
23	Bromophos-methyl (Bromophos)	13.76	0.9965	104	1.5	0.9985	93	4.0
24	Bromopropylate	20.38	0.9965	106	1.8	0.9985	93	1.7
25	Bupirimate	16.35	0.9971	103	2.1	NA	NA	NA
26	Carbophenothion	18.31	0.9928	108	2.2	0.9990	93	3.9
27	Carfentrazon-ethyl	18.23	0.9957	111	1.8	0.9989	91	2.7
28	Chlorbenside	15.02	0.9989	97	2.0	0.9981	83	3.0
29	Chlordane alpha-Cis	15.48	0.9973	110	5.6	0.9982	91	4.3
30	Chlordane Gamma-trans	15.07	0.9944	101	1.7	0.9978	94	2.8
31	Chlorfenapyr	16.64	0.9977	99	4.4	0.9983	88	10.3
32	Chlorfenson	15.75	0.9978	93	2.2	0.9981	94	2.8
33	Chlorfenvinphos	14.36	0.9928	109	2.6	0.9981	96	3.5
34	Chlorobenzilate	17.17	0.9980	101	3.8	0.9983	89	3.1
35	Chloroneb	8.07	0.9962	92	3.1	0.9977	95	2.4
36	Chlorpropham	9.35	0.9986	102	4.1	0.9986	84	4.1
37	Chlorpyrifos-ethyl	13.13	0.9972	104	2.0	0.9986	99	2.9
38	Chlorpyrifos-methyl	11.90	0.9920	108	3.0	0.9972	91	4.2
39	Chlorthal-dimethyl (Dacthal)	13.30	0.9972	95	2.7	0.9990	96	2.6
40	Chlorothalonil	10.95	NA	NA	NA	0.9952	100	6.0
41	Chlorthiophos	17.54	0.9985	100	1.0	0.9982	90	1.4
42	Chlozolinate	14.27	0.9954	99	3.6	0.9989	96	4.1

<LOQ= Below limit of quantification (0.01 mg/kg)

Appendix 1 (continued). List of pesticides analyzed in rice and wheat results (retention time, linearity, recovery, and precision at 0.01 mg/kg) with the µSPE clean-up

			Rice (0.01 mg/kg)			Wheat (0.01 mg/kg)		
Sr. No.	Compound Name	RT	R^2	%Rec.	%RSD	R^2	%Rec.	%RSD
43	Clomazone	10.42	0.9980	104	2.0	0.9988	92	2.1
44	Coumaphos	23.84	0.9965	107	1.4	0.9982	94	3.1
45	Cycloate	9.22	0.9973	88	4.9	0.9940	103	6.2
46	Cyfluthrin peak 1	24.71	0.9984	109	3.1	0.9981	95	3.0
47	Cyfluthrin peak 2	24.91	0.9979	113	2.3	0.9982	98	2.5
48	Cyfluthrin peak 3	25.02	0.9988	108	2.9	0.9974	97	4.1
49	Cyfluthrin peak 4	25.11	0.9985	107	3.6	0.9978	94	2.0
50	Cyhalothrin I (lambda)	22.20	0.9969	108	2.4	0.9988	95	5.8
51	Cypermethrin peak 1	25.31	0.9984	104	4.4	0.9980	94	3.1
52	Cypermethrin peak 2	25.52	0.9978	109	3.9	0.9984	97	4.2
53	Cypermethrin peak 3	25.62	0.9985	109	2.9	0.9979	95	4.0
54	Cypermethrin peak 4	25.71	0.9978	111	4.2	0.9969	96	4.8
55	Cyprodinil	14.11	0.9988	102	2.3	0.9959	92	2.0
56	DDD p,p	17.45	0.9983	103	2.0	0.9991	96	3.0
57	DDD, o, p	16.34	0.9983	97	2.3	0.9988	95	2.5
58	DDE o,p	15.14	0.9982	93	1.9	0.9984	93	2.2
59	DDE p, p	16.13	0.9978	88	1.7	0.9983	92	2.6
60	DDT o,p	17.55	0.9972	97	2.4	0.9980	91	2.4
61	DDT p,p	18.70	0.9971	98	3.2	0.9982	90	2.7
62	Deltamethrin	28.25	0.9962	112	3.6	0.9968	99	4.8
63	Diallate-cis	9.95	0.9981	103	4.2	0.9989	93	4.6
64	Diallate-trans	9.74	0.9974	104	2.3	0.9984	93	4.0
65	Diazinon	10.71	0.9970	110	3.8	0.9981	94	3.9
66	Dichlobenil	6.90	0.9981	91	1.6	0.9989	94	1.4
67	Dichlorobenzophenone, 4, 4	13.56	0.9981	96	2.0	0.9983	92	0.9
68	Dicloran (Bortran)	10.15	0.9989	112	6.5	0.9958	94	1.8
69	Dieldrin	16.31	0.9965	94	4.8	0.9968	100	5.6
70	Dimethachlor	11.75	0.9956	116	1.5	0.9988	93	2.9
71	Diphenamid	13.75	0.9971	103	2.0	0.9992	90	2.9
72	Diphenylamine	9.13	0.9982	100	8.1	0.9982	78	5.6
73	Disulfoton	11.01	0.9942	111	1.5	0.9981	78	3.8
74	Edifenphos	18.38	0.9990	104	3.7	0.9961	104	3.9
75	Endosulfan ether	11.67	0.9978	97	2.0	0.9987	93	2.3
76	Endosulfan peak 1	15.48	0.9926	113	8.2	0.9977	93	8.5
77	Endosulfan peak 2	17.27	0.9966	93	5.0	0.9992	91	3.3
78	Endosulfan sulfate	18.51	0.9962	96	2.5	0.9985	104	4.3
79	Endrin	16.95	0.9971	106	2.4	0.9985	93	6.9
80	Endrin Aldehyde	17.75	NA	NA	NA	0.9935	100	6.7
81	Endrin-Ketone	20.05	0.9981	92	4.4	0.9979	92	6.4
82	EPN	20.31	0.9962	106	1.8	0.9972	96	2.6
83	Esfenvalerate	27.35	0.9992	104	5.5	0.9979	92	3.1
84	Ethalfluralin	9.23	0.9983	89	11.4	0.9986	92	7.5
85	Ethion	17.45	0.9943	107	1.9	0.9985	92	2.1

<LOQ= Below limit of quantification (0.01 mg/kg)

Appendix 1 (continued). List of pesticides analyzed in rice and wheat results (retention time, linearity, recovery, and precision at 0.01 mg/kg) with the µSPE clean-up

			Rice (0.01 mg/kg)			Wheat (0.01 mg/kg)		
Sr. No.	Compound Name	RT	R^2	%Rec.	%RSD	R^2	%Rec.	%RSD
86	Etofenprox	25.91	0.9979	104	1.5	0.9978	91	4.1
87	Etridiazole (Terrazole)	7.66	0.9967	108	2.7	0.9986	91	1.3
88	Fenamiphos	15.69	0.9949	110	5.1	NA	NA	NA
89	Fenarimol	22.44	0.9979	111	2.1	0.9985	90	2.4
90	Fenchlorfos	12.35	0.9976	109	2.0	0.9983	91	2.8
91	Fenitrothion	12.68	0.9932	107	2.6	0.9973	90	2.1
92	Fenpropathrin	20.67	0.9981	104	2.7	0.9986	99	3.3
93	Fenson	13.71	0.9975	99	2.0	0.9983	93	1.9
94	Fenthion	13.23	0.9964	100	2.2	0.9982	70	1.3
95	Fenvalerate	26.98	0.9947	114	1.4	0.9972	98	2.7
96	Fipronil	14.17	0.9968	115	4.8	0.9971	93	2.7
97	Fluazifop-P-butyl	16.90	0.9983	102	2.5	0.9990	96	2.7
98	Fluchloralin	10.75	0.9909	113	4.4	0.9951	94	3.9
99	Flucythrinate peak 1	25.65	0.9969	108	1.4	0.9983	99	1.7
100	Flucythrinate peak 2	26.03	0.9988	113	4.2	0.9980	99	2.0
101	Fludioxonil	15.80	0.9983	93	3.2	0.9956	95	7.2
102	Fluquinconazole	23.86	0.9976	105	3.5	0.9986	95	2.2
103	Fluridone	26.27	0.9931	113	2.1	0.9963	92	2.1
104	Flusilazole	16.32	0.9973	109	2.6	1.0000	92	13.1
105	Flutolanil	15.71	0.9975	95	3.4	0.9995	94	9.1
106	Flutriafol	15.55	0.9981	105	2.8	0.9975	98	3.2
107	Fluvalinate peak 1	27.25	0.9977	114	2.1	0.9972	102	3.9
108	Fluvalinate peak 2	27.38	0.9934	117	3.1	0.9954	103	3.3
109	Fonofos	10.75	0.9957	115	4.5	0.9970	76	8.8
110	Heptachlor	12.35	0.9975	108	2.2	0.9984	93	3.0
111	Heptachlor epoxide	14.38	0.9969	103	3.7	0.9989	94	5.5
112	Hexachlorobenzene	10.03	0.9978	80	17.8	0.9968	99	10.3
113	Hexazinone	18.81	0.9970	107	2.4	0.9984	89	1.4
114	Iodofenfos	15.76	0.9931	104	2.3	0.9975	93	3.8
115	Iprodione	20.02	0.9929	104	7.0	NA	NA	NA
116	Isazophos	11.00	0.9932	118	4.4	0.9970	90	4.9
117	Isodrin	14.12	0.9984	86	5.2	0.9975	90	3.2
118	Isopropalin	13.87	0.9929	109	2.0	0.9957	92	3.7
119	Lenacil	18.50	0.9973	100	4.4	0.9984	90	1.5
120	Leptophos	21.50	0.9991	99	2.4	0.9965	92	2.3
121	Linuron	12.88	NA	NA	NA	0.9985	108	10.8
122	Malathion	12.90	0.9954	109	1.8	0.9980	84	4.5
123	Metalaxyl	12.24	0.9958	113	3.2	0.9979	94	4.3
124	Metazachlor	14.12	0.9976	115	3.4	0.9976	94	3.3
125	Methacrifos	7.95	0.9969	106	1.4	0.9986	93	2.9
126	Methoxychlor	20.56	0.9959	107	3.0	0.9984	93	1.8
127	Metolachlor	13.09	0.9962	102	2.3	0.9987	93	2.1
128	Mevinphos	7.42	0.9975	96	4.0	0.9993	74	2.2

<LOQ= Below limit of quantification (0.01 mg/kg)

Appendix 1 (continued). List of pesticides analyzed in rice and wheat results (retention time, linearity, recovery, and precision at 0.01 mg/kg) with the µSPE clean-up

			Rice (0.01 mg/kg)			Wheat (0.01 mg/kg)		
Sr. No.	Compound Name	RT	R^2	%Rec.	%RSD	R^2	%Rec.	%RSD
129	MGK-264 A	13.80	0.9983	96	3.8	0.9983	92	7.2
130	MGK-264 B	14.16	0.9975	99	3.9	0.9989	97	6.7
131	Mirex	22.20	0.9996	86	7.0	0.9957	81	5.7
132	Myclobutanil	16.23	0.9973	109	2.5	NA	NA	NA
133	N-(2,4-Dimethylphenyl)formamide	7.89	0.9963	112	7.1	0.9983	78	5.3
134	Nitralin	19.45	0.9944	107	4.0	0.9925	100	8.4
135	Nitrofen	16.87	0.9954	103	3.9	0.9978	92	1.9
136	Nonachlor-cis	17.47	0.9976	99	3.1	0.9980	92	2.5
137	Nonachlor-trans	15.58	0.9949	105	4.4	0.9963	89	4.6
138	Norflurazon	18.35	0.9971	107	1.5	0.9981	91	0.9
139	Ortho-phenylphenol	8.23	0.9994	96	1.6	0.9984	75	3.5
140	Oxadiazon	16.14	0.9972	93	2.6	0.9986	96	3.1
141	Oxyfluorfen	16.30	0.9929	110	4.3	0.9966	94	4.5
142	Paclobutrazol	15.21	0.9951	113	4.6	0.9979	89	3.5
143	Parathion (ethyl)	13.33	0.9959	108	3.4	0.9973	91	3.3
144	Parathion-methyl	12.04	0.9920	119	1.4	0.9982	90	3.5
145	Pebulate	7.71	0.9964	115	7.9	0.9989	103	2.5
146	Penconazole	14.25	0.9979	108	2.9	0.9986	92	1.3
147	Pendimethalin	14.08	0.9901	113	3.0	0.9951	91	5.8
148	Pentachloroaniline	11.64	0.9986	109	7.7	0.9974	101	4.6
149	Pentachloroanisole	10.12	0.9984	95	1.4	0.9987	90	3.6
150	Pentachlorobenzene	8.27	0.9977	88	11.0	0.9985	103	7.0
151	Pentachlorobenzonitrile	10.60	0.9958	104	3.1	0.9973	92	3.0
152	Pentachlorothioanisole	12.91	0.9951	87	4.2	0.9965	88	5.4
153	Permethrin peak 1	23.66	0.9983	78	7.4	0.9988	100	3.3
154	Permethrin peak 2	23.91	0.9980	110	5.0	0.9982	90	2.2
155	Perthane (Ethylan)	16.92	0.9975	103	2.1	0.9992	94	1.8
156	Phenothrin	21.38	0.9990	101	12.9	0.9985	105	11.3
157	Phorate	9.75	0.9975	117	2.9	0.9979	85	3.4
158	Phosalone	21.45	0.9905	108	1.2	0.9981	95	3.2
159	Phosmet	20.17	0.9958	101	2.3	0.9987	90	2.3
160	Phthalimide	7.80	0.9972	106	13.0	0.9989	87	5.6
161	Piperonyl butoxide	19.45	0.9945	107	2.8	0.9982	93	3.1
162	Pirimiphos-ethyl	13.71	0.9978	108	2.8	0.9984	94	1.9
163	Pirimiphos-methyl	12.60	0.9979	109	1.2	0.9991	93	5.6
164	Pretilachlor	15.93	0.9976	97	2.0	0.9982	93	4.2
165	Prochloraz	24.00	0.9936	112	4.2	0.9967	91	3.2
166	Procymidone	14.65	0.9968	104	3.6	0.9985	91	1.8
167	Prodiamine	12.64	0.9921	111	4.2	0.9972	92	1.7
168	Profenofos	15.99	0.9947	102	4.1	0.9973	103	6.1
169	Profluralin	10.49	0.9917	107	2.7	0.9987	89	4.4

<LOQ= Below limit of quantification (0.01 mg/kg)

Appendix 1 (continued). List of pesticides analyzed in rice and wheat results (retention time, linearity, recovery, and precision at 0.01 mg/kg) with the µSPE clean-up

			Rice (0.01 mg/kg)			Wheat (0.01 mg/kg)		
Sr. No.	Compound Name	RT	R^2	%Rec.	%RSD	R^2	%Rec.	%RSD
170	Propachlor	8.94	0.9987	104	1.9	0.9990	96	3.2
171	Propanil	11.79	0.9945	114	7.4	0.9977	90	5.4
172	Propargite	19.26	0.9978	102	4.3	0.9991	92	9.2
173	Propisochlor	12.19	0.9958	112	2.5	0.9993	94	2.5
174	Propyzamide	10.70	0.9973	109	1.5	0.9987	91	2.6
175	Prothiofos	15.86	0.9982	93	3.4	0.9947	90	9.7
176	Pyraclofos	22.99	0.9946	97	4.1	0.9969	99	3.1
177	Pyrazophos	22.50	0.9931	117	2.0	0.9979	93	2.9
178	Pyridaben	23.91	0.9965	108	1.8	0.9979	96	2.2
179	Pyridaphenthion	19.97	0.9927	110	1.7	0.9987	93	2.9
180	Pyrimethanil	10.88	0.9985	110	4.0	0.9975	92	2.5
181	Pyriproxyfen	21.80	0.9981	105	1.7	0.9981	90	3.0
182	Quinalphos	14.54	0.9931	104	2.5	0.9969	91	7.5
183	Quintozene	10.52	0.9965	101	3.9	0.9983	89	3.1
184	Resmethrin peak 2	19.54	0.9982	98	3.2	0.9988	101	3.9
185	Sulfotep	9.48	0.9948	112	3.1	0.9974	93	1.4
186	Sulprofos	17.97	0.9982	101	2.5	0.9987	76	2.1
187	Tebuconazole	19.13	0.9933	109	3.1	0.9981	89	2.4
188	Tebufenpyrad	20.85	0.9992	101	1.2	0.9987	96	1.6
189	Tecnazene	8.85	0.9994	98	3.2	0.9990	89	4.5
190	Tefluthrin	11.01	0.9960	104	2.1	0.9985	92	3.2
191	Terbacil	11.00	0.9995	106	4.9	0.9974	87	5.0
192	Terbufos	10.62	0.9907	115	1.2	0.9975	88	1.7
193	Terbuthylazine	10.61	0.9984	103	2.9	0.9991	92	4.4
194	Tetrachlorvinphos	15.16	0.9910	101	3.3	0.9984	105	6.4
195	Tetradifon	21.24	0.9984	93	3.2	0.9985	93	1.2
196	Tetrahydrophthalimide (THPI)	7.97	0.9991	105	5.3	0.9986	97	2.9
197	Tetramethrin peak 1	20.13	0.9965	85	10.7	0.9966	115	9.1
198	Tetramethrin peak 2	20.42	0.9968	110	3.8	0.9986	96	2.6
199	Tolclofos-methyl	12.08	0.9968	110	2.9	0.9983	91	2.0
200	Tolyfluanid	14.34	NA	NA	NA	0.9991	69	3.9
201	Transfluthrin	12.10	0.9965	108	3.7	0.9979	96	3.5
202	Triadimefon	13.42	0.9967	107	4.0	0.9987	92	5.7
203	Triadimenol	14.65	0.9980	106	2.6	0.9978	98	6.8
204	Triallate	11.20	0.9966	108	2.3	0.9985	94	3.0
205	Triazophos	17.91	0.9915	117	1.7	0.9988	94	5.2
206	Triflumizole	14.71	0.9976	116	7.4	0.9988	91	9.4
207	Trifluralin	9.37	0.9987	116	1.5	0.9975	91	3.0
208	Vinclozolin	11.98	0.9952	103	2.8	0.9971	97	3.3
209	Tricyclazole	15.97	0.9964	116	8.5	NA	NA	NA
210	Triphenylphosphate (IS)	19.28	NA	NA	NA	NA	NA	NA

<LOQ= Below limit of quantification (0.01 mg/kg)

Appendix 2. Target list of analytes with their SRM parameters

Compound Name	RT (min)	Q1	Q3	CE
2,3,5,6-Tetrachloroaniline	9.19	230.8	157.9	18
2,3,5,6-Tetrachloroaniline	9.19	230.8	159.8	18
2,3,5,6-Tetrachloroaniline	9.19	230.8	194.8	10
2,4'-Methoxychlor	18.91	227.1	121.1	10
2,4'-Methoxychlor	18.91	228.1	122.1	16
2,4'-Methoxychlor	18.91	152.0	126.1	24
2,4'-Methoxychlor	18.91	152.0	151.1	16
3,4-Dichloroaniline	7.56	160.9	99.0	20
3,4-Dichloroaniline	7.56	160.9	90.0	18
3,4-Dichloroaniline	7.56	160.9	126.0	10
4,4'-Methoxychlor olefin	18.32	238.1	152.1	34
4,4'-Methoxychlor olefin	18.32	238.1	223.1	10
4,4'-Methoxychlor olefin	18.32	308.0	238.2	12
Acetochlor	11.86	146.1	130.0	24
Acetochlor	11.86	146.1	131.0	12
Acetochlor	11.86	174.1	146.1	10
Acetochlor	11.86	223.1	132.0	20
Acetochlor	11.86	131.8	117.0	14
Acetochlor	11.86	146.0	117.7	8
Acrinathrin	22.59	208.1	180.9	8
Acrinathrin	22.59	289.0	93.1	8
Acrinathrin	22.59	181.0	152.0	22
Alachlor	12.09	188.1	130.0	32
Alachlor	12.09	188.1	132.0	14
Alachlor	12.09	188.1	160.1	8
Alachlor	12.09	160.1	131.7	10
Aldrin	13.32	254.9	219.9	20
Aldrin	13.32	262.7	191.0	30
Aldrin	13.32	262.7	192.9	32
Aldrin	13.32	330.0	298.9	10
Allidochlor	6.48	132.0	56.1	8
Allidochlor	6.48	134.0	56.0	8
Allidochlor	6.48	132.0	49.0	26
Allidochlor	6.48	138.1	95.9	6
Anthraquinone	13.33	180.1	152.0	12
Anthraquinone	13.33	208.1	152.0	22
Anthraquinone	13.33	208.1	180.1	10
Atrazine	10.32	200.1	122.0	8
Atrazine	10.32	200.1	132.0	8
Atrazine	10.32	215.1	58.1	10
Azinphos-ethyl	22.67	132.0	77.0	12
Azinphos-ethyl	22.67	132.0	51.0	26
Azinphos-ethyl	22.67	160.0	77.0	16
Azinphos-methyl	21.52	132.0	77.0	12
Azinphos-methyl	21.52	160.0	50.9	34
Azinphos-methyl	21.52	160.0	77.0	16
Benfluralin	9.42	292.0	264.0	8
Benfluralin	9.42	292.0	160.0	20
Benfluralin	9.42	292.0	206.0	12
BHC, Alpha	9.91	180.9	144.9	14
BHC, Alpha	9.91	216.9	181.0	8
BHC, Alpha	9.91	218.9	183.0	8
BHC, Alpha	9.91	182.8	146.7	12
BHC, Alpha	9.91	218.8	146.6	20
BHC, Beta	10.38	180.9	145.0	14
BHC, Beta	10.38	216.9	180.9	8
BHC, Beta	10.38	218.9	183.0	8
BHC, Beta	10.38	218.7	146.6	18
BHC, delta	11.16	180.9	144.9	14
BHC, delta	11.16	182.9	147.0	14
BHC, delta	11.16	218.9	182.9	8
BHC, delta	11.16	218.8	146.5	20
BHC, gamma	10.60	180.9	144.9	12
BHC, gamma	10.60	216.9	180.9	8
BHC, gamma	10.60	218.9	183.0	8
BHC, gamma	10.60	180.9	109.0	26
Bifenthrin	20.37	181.0	165.9	10
Bifenthrin	20.37	181.0	179.0	12
Bifenthrin	20.37	165.1	163.6	24
Bromfenvinphos	15.60	266.9	159.0	14
Bromfenvinphos	15.60	268.9	161.1	14
Bromfenvinphos	15.60	323.1	266.9	10
Bromfenvinphos	15.60	266.9	203.0	10
Bromfenvinphos-methyl	14.38	294.9	109.0	16
Bromfenvinphos-methyl	14.38	294.9	79.1	30
Bromfenvinphos-methyl	14.38	109.0	79.0	6
Bromophos-ethyl	14.99	358.8	302.8	14
Bromophos-ethyl	14.99	302.8	284.8	14
Bromophos-ethyl	14.99	302.8	210.9	30
Bromophos-ethyl	14.99	96.9	65.0	16
Bromophos-ethyl	14.99	96.9	78.9	12
Bromophos-methyl (Bromophos)	13.77	330.8	315.8	14
Bromophos-methyl (Bromophos)	13.77	328.9	313.8	14
Bromophos-methyl (Bromophos)	13.77	330.8	93.0	24
Bromophos-methyl (Bromophos)	13.77	125.0	79.0	6
Bromopropylate	20.39	340.8	185.0	14

Appendix 2 (continued). Target list of analytes with their SRM parameters

Compound Name	RT (min)	Q1	Q3	CE
Bromopropylate	20.39	184.9	156.9	12
Bromopropylate	20.39	184.9	75.5	30
Bupirimate	16.36	273.1	193.2	8
Bupirimate	16.36	273.1	108.0	14
Bupirimate	16.36	316.2	208.1	10
Bupirimate	16.36	208.1	140.1	12
Bupirimate	16.36	208.1	165.0	12
Captafol	19.38	150.1	79.0	6
Captafol	19.38	151.1	79.1	18
Captafol	19.38	183.1	79.1	8
Captafol	19.38	150.1	77.2	24
Captan	14.58	149.0	70.0	20
Captan	14.58	117.0	82.0	30
Captan	14.58	151.0	79.0	14
Captan	14.58	151.0	80.0	6
Captan	14.58	149.0	78.8	14
Captan	14.58	149.0	105.0	6
Carbophenothion	18.31	342.0	157.0	10
Carbophenothion	18.31	157.0	45.0	12
Carbophenothion	18.31	199.0	142.9	10
Carfentrazon-ethyl	18.23	340.1	312.1	10
Carfentrazon-ethyl	18.23	290.0	99.9	36
Carfentrazon-ethyl	18.23	311.9	150.7	18
Chlorbenside	15.03	125.0	89.0	16
Chlorbenside	15.03	125.0	99.0	16
Chlorbenside	15.03	268.0	125.0	10
Chlorbenside	15.03	125.0	62.8	28
Chlordane alpha-Cis	15.49	372.8	265.9	14
Chlordane alpha-Cis	15.49	271.8	236.8	12
Chlordane alpha-Cis	15.49	271.8	236.8	14
Chlordane alpha-Cis	15.49	372.8	265.8	20
Chlordane alpha-Cis	15.49	374.7	265.8	20
Chlordane alpha-Cis	15.49	376.6	268.0	20
Chlordane Gamma-trans	15.08	372.8	265.9	20
Chlordane Gamma-trans	15.08	374.8	265.9	20
Chlordane Gamma-trans	15.08	271.9	236.9	14
Chlordane Gamma-trans	15.08	372.7	263.7	20
Chlorfenapyr	16.64	136.9	102.0	12
Chlorfenapyr	16.64	248.9	112.0	24
Chlorfenapyr	16.64	248.9	137.1	18
Chlorfenapyr	16.64	327.9	246.9	14
Chlorfenson	15.76	175.0	111.0	10
Chlorfenson	15.76	111.0	75.0	14
Chlorfenson	15.76	175.0	75.0	28
Chlorfenvinphos	14.37	266.9	159.0	16
Chlorfenvinphos	14.37	268.9	161.0	14
Chlorfenvinphos	14.37	323.0	266.9	14
Chlorfenvinphos	14.37	266.9	203.0	10
Chlorobenzilate	17.18	139.0	111.0	12
Chlorobenzilate	17.18	251.0	111.0	34
Chlorobenzilate	17.18	251.0	139.0	14
Chlorobenzilate	17.18	111.0	75.1	14
Chlorobenzilate	17.18	139.0	74.9	26
Chlorobenzilate	17.18	139.0	111.0	12
Chloroneb	8.00	190.9	113.0	14
Chloroneb	8.00	193.0	53.1	32
Chloroneb	8.00	193.0	115.0	14
Chloroneb	8.00	190.9	141.0	10
Chloroneb	8.00	206.0	190.9	12
Chlorothalonil	10.95	263.9	132.9	40
Chlorothalonil	10.95	265.9	133.0	36
Chlorothalonil	10.95	265.9	170.0	24
Chlorothalonil	10.95	228.8	168.0	8
Chlorpropham	9.35	127.0	65.0	20
Chlorpropham	9.35	171.0	127.0	8
Chlorpropham	9.35	213.0	127.0	14
Chlorpropham	9.35	213.0	171.0	6
Chlorpyrifos-ethyl	13.14	198.9	171.0	14
Chlorpyrifos-ethyl	13.14	196.9	168.9	12
Chlorpyrifos-ethyl	13.14	313.9	257.9	12
Chlorpyrifos-ethyl	13.14	196.7	107.0	36
Chlorpyrifos-methyl	11.91	285.9	270.9	14
Chlorpyrifos-methyl	11.91	285.9	92.9	20
Chlorpyrifos-methyl	11.91	287.9	92.9	20
Chlorpyrifos-methyl	11.91	287.9	272.9	14
Chlorpyrifos-methyl	11.91	125.0	47.0	12
Chlorpyrifos-methyl	11.91	125.0	79.0	6
Chlorthal-dimethyl (Dacthal)	13.30	300.9	272.9	12
Chlorthal-dimethyl (Dacthal)	13.30	300.9	222.9	22
Chlorthal-dimethyl (Dacthal)	13.30	222.9	166.9	20
Chlorthiophos	17.55	324.9	268.9	12
Chlorthiophos	17.55	268.9	205.0	14
Chlorthiophos	17.55	296.9	268.9	8
Chlozolinate	14.28	186.0	145.0	14
Chlozolinate	14.28	188.0	147.0	14
Chlozolinate	14.28	259.0	187.9	12

Appendix 2 (continued). Target list of analytes with their SRM parameters

Compound Name	RT (min)	Q1	Q3	CE
Chlozolinate	14.28	259.0	152.9	26
Chlozolinate	14.28	331.0	259.0	8
Clomazone	10.42	125.0	89.0	16
Clomazone	10.42	125.0	99.0	16
Clomazone	10.42	204.0	107.0	18
Clomazone	10.42	138.0	74.9	24
Clomazone	10.42	138.0	111.0	12
Coumaphos	23.85	226.0	163.0	18
Coumaphos	23.85	362.0	226.0	10
Coumaphos	23.85	209.9	119.0	22
Coumaphos	23.85	209.9	182.0	10
Cycloate	9.22	154.1	83.1	8
Cycloate	9.22	154.1	55.1	18
Cycloate	9.22	215.1	154.1	6
Cycloate	9.22	83.1	55.1	6
Cyfluthrin peak 1	24.70	163.0	127.1	6
Cyfluthrin peak 1	24.70	226.0	206.1	12
Cyfluthrin peak 1	24.70	163.0	91.1	12
Cyfluthrin peak 1	24.70	163.0	65.1	26
Cyfluthrin peak 2	24.91	163.0	127.0	6
Cyfluthrin peak 2	24.91	226.0	206.1	12
Cyfluthrin peak 2	24.91	163.0	91.1	12
Cyfluthrin peak 2	24.91	206.0	151.1	18
Cyfluthrin peak 3	25.02	163.0	127.0	6
Cyfluthrin peak 3	25.02	226.0	206.1	12
Cyfluthrin peak 3	25.02	163.0	91.1	12
Cyfluthrin peak 4	25.11	163.0	127.0	6
Cyfluthrin peak 4	25.11	226.0	206.1	10
Cyfluthrin peak 4	25.11	163.0	91.1	12
Cyhalothrin I (lambda)	22.21	180.9	152.0	22
Cyhalothrin I (lambda)	22.21	197.1	141.1	10
Cyhalothrin I (lambda)	22.21	207.9	180.9	8
Cypermethrin peak 1	25.32	163.0	127.1	6
Cypermethrin peak 1	25.32	165.0	127.1	5
Cypermethrin peak 1	25.32	163.0	91.1	12
Cypermethrin peak 1	25.32	180.9	152.1	20
Cypermethrin peak 2	25.53	163.0	127.0	6
Cypermethrin peak 2	25.53	165.0	127.1	5
Cypermethrin peak 2	25.53	163.0	91.1	12
Cypermethrin peak 2	25.53	180.9	151.9	18
Cypermethrin peak 3	25.61	163.0	127.0	6
Cypermethrin peak 3	25.61	165.0	127.1	5
Cypermethrin peak 3	25.61	163.0	91.0	12
Cypermethrin peak 3	25.61	180.9	152.2	20
Cypermethrin peak 4	25.72	163.0	127.1	6
Cypermethrin peak 4	25.72	165.0	127.1	5
Cypermethrin peak 4	25.72	163.0	91.1	12
Cypermethrin peak 4	25.72	180.9	152.2	20
Cyprodinil	14.12	224.1	208.1	18
Cyprodinil	14.12	224.1	197.1	20
Cyprodinil	14.12	225.1	210.1	16
DDD p,p	17.46	235.0	165.0	20
DDD p,p	17.46	235.0	199.0	14
DDD p,p	17.46	237.0	165.0	20
DDD, o, p	16.34	235.0	165.0	20
DDD, o, p	16.34	235.0	199.0	14
DDD, o, p	16.34	237.0	165.0	20
DDE o,p	15.14	246.0	176.1	28
DDE o,p	15.14	248.0	176.1	30
DDE o,p	15.14	317.9	248.0	18
DDE o,p	15.14	317.8	246.0	20
DDE p, p	16.14	246.0	176.1	28
DDE p, p	16.14	315.9	246.0	14
DDE p, p	16.14	317.9	246.0	20
DDE p, p	16.14	317.9	248.0	18
DDT o,p	17.56	235.0	165.1	22
DDT o,p	17.56	235.0	199.1	10
DDT o,p	17.56	237.0	165.1	22
DDT p,p	18.70	235.0	165.1	22
DDT p,p	18.70	236.8	165.0	22
DDT p,p	18.70	235.0	199.5	10
Deltamethrin	28.25	252.8	92.9	16
Deltamethrin	28.25	181.0	152.1	22
Deltamethrin	28.25	252.8	172.0	8
Diallate-cis	9.94	234.1	150.0	18
Diallate-cis	9.94	235.8	152.0	18
Diallate-cis	9.94	235.8	194.0	12
Diallate-trans	9.75	234.1	150.0	18
Diallate-trans	9.75	234.1	192.0	12
Diallate-trans	9.75	235.8	152.0	18
Diallate-trans	9.75	235.8	194.0	12
Diazinon	10.72	137.1	84.1	12
Diazinon	10.72	137.1	54.1	20
Diazinon	10.72	199.0	92.9	14
Diazinon	10.72	179.1	121.5	26
Dichlobenil	6.90	170.9	99.9	24

Appendix 2 (continued). Target list of analytes with their SRM parameters

Compound Name	RT (min)	Q1	Q3	CE
Dichlobenil	6.90	170.9	136.0	12
Dichlobenil	6.90	172.8	99.8	24
Dichlorobenzophenone, 4, 4	13.57	139.0	111.0	12
Dichlorobenzophenone, 4, 4	13.57	139.0	74.9	26
Dichlorobenzophenone, 4, 4	13.57	141.0	113.0	10
Dichlorobenzophenone, 4, 4	13.57	111.0	74.9	12
Dicloran (Bortran)	10.16	206.0	176.0	10
Dicloran (Bortran)	10.16	160.0	124.1	8
Dicloran (Bortran)	10.16	176.0	148.0	12
Dieldrin	16.31	262.9	192.9	30
Dieldrin	16.31	262.9	190.9	30
Dieldrin	16.31	262.9	227.8	16
Dieldrin	16.31	276.9	240.8	6
Dimethachlor	11.76	197.1	148.0	10
Dimethachlor	11.76	134.0	77.0	24
Dimethachlor	11.76	134.0	105.1	12
Diphenamid	13.75	167.1	152.1	16
Diphenamid	13.75	167.1	165.1	20
Diphenamid	13.75	239.1	167.1	8
Diphenamid	13.75	239.1	72.1	10
Diphenylamine	9.14	168.1	167.1	14
Diphenylamine	9.14	169.1	167.1	24
Diphenylamine	9.14	169.1	168.1	12
Diphenylamine	9.14	168.1	139.0	38
Disulfoton	11.01	88.0	59.8	6
Disulfoton	11.01	88.0	45.0	18
Disulfoton	11.01	142.0	81.0	10
Disulfoton	11.01	185.9	96.9	16
Edifenphos	18.39	172.9	109.0	8
Edifenphos	18.39	310.0	109.0	26
Edifenphos	18.39	172.9	65.1	30
Endosulfan ether	11.68	238.9	204.0	12
Endosulfan ether	11.68	240.9	206.0	14
Endosulfan peak 1	15.49	240.8	205.8	14
Endosulfan peak 1	15.49	262.8	192.9	30
Endosulfan peak 1	15.49	194.9	160.0	8
Endosulfan peak 1	15.49	194.7	125.0	22
Endosulfan peak 1	15.49	194.7	159.4	8
Endosulfan peak 2	17.28	158.9	123.0	12
Endosulfan peak 2	17.28	240.6	205.8	12
Endosulfan peak 2	17.28	194.9	159.0	8
Endosulfan peak 2	17.28	236.8	118.9	30
Endosulfan sulfate	18.52	271.7	236.8	12
Endosulfan sulfate	18.52	238.7	203.9	12
Endosulfan sulfate	18.52	271.7	234.9	12
Endrin	16.97	262.8	192.9	30
Endrin	16.97	244.9	173.0	22
Endrin	16.97	280.8	244.9	8
Endrin Aldehyde	17.75	173.0	138.1	16
Endrin Aldehyde	17.75	249.8	214.9	24
Endrin Aldehyde	17.75	278.9	242.9	10
Endrin Aldehyde	17.75	344.9	281.0	8
Endrin-Ketone	20.06	316.8	281.0	10
Endrin-Ketone	20.06	316.8	208.9	28
Endrin-Ketone	20.06	209.2	138.4	30
EPN	20.31	169.0	77.0	22
EPN	20.31	157.0	77.0	22
EPN	20.31	169.0	141.0	8
Esfenvalerate	27.36	167.0	125.0	10
Esfenvalerate	27.36	125.0	89.0	18
Esfenvalerate	27.36	167.0	89.0	32
Esfenvalerate	27.36	225.1	119.1	18
Ethalfluralin	9.24	276.0	202.0	14
Ethalfluralin	9.24	276.0	248.1	8
Ethalfluralin	9.24	315.9	276.1	8
Ethalfluralin	9.24	292.0	264.0	8
Ethion	17.45	230.9	128.9	22
Ethion	17.45	230.9	174.9	12
Ethion	17.45	153.0	97.0	10
Ethion	17.45	120.9	65.0	10
Etofenprox	25.92	163.1	107.1	16
Etofenprox	25.92	163.1	135.1	10
Etofenprox	25.92	163.1	77.1	32
Etridiazole (Terrazole)	7.67	182.8	139.9	14
Etridiazole (Terrazole)	7.67	211.0	139.9	18
Etridiazole (Terrazole)	7.67	211.0	182.9	10
Fenamiphos	15.60	303.1	195.0	8
Fenamiphos	15.60	154.0	139.0	10
Fenamiphos	15.60	217.0	202.0	12
Fenarimol	22.45	139.0	111.0	14
Fenarimol	22.45	139.0	74.9	26
Fenarimol	22.45	219.0	107.0	10
Fenchlorfos	12.35	284.9	269.9	14
Fenchlorfos	12.35	284.9	93.0	24
Fenchlorfos	12.35	286.9	271.9	14
Fenchlorfos	12.35	124.9	47.0	12

Appendix 2 (continued). Target list of analytes with their SRM parameters

Compound Name	RT (min)	Q1	Q3	CE
Fenchlorfos	12.35	124.9	79.0	6
Fenchlorfos	12.35	169.0	110.4	6
Fenitrothion	12.69	277.0	260.0	6
Fenitrothion	12.69	277.0	109.0	16
Fenitrothion	12.69	277.0	109.0	14
Fenitrothion	12.69	125.0	79.0	8
Fenpropathrin	20.67	181.0	151.9	22
Fenpropathrin	20.67	181.0	126.8	28
Fenpropathrin	20.67	97.1	55.1	6
Fenson	13.71	141.0	77.0	8
Fenson	13.71	141.0	50.9	30
Fenson	13.71	268.0	77.0	20
Fenson	13.71	77.0	51.0	14
Fenthion	13.24	278.0	109.0	18
Fenthion	13.24	278.0	169.0	14
Fenthion	13.24	278.0	125.0	14
Fenthion	13.24	245.3	125.0	12
Fenvalerate	26.98	167.0	125.0	10
Fenvalerate	26.98	125.0	89.0	18
Fenvalerate	26.98	167.0	89.0	32
Fipronil	14.18	366.9	212.9	28
Fipronil	14.18	366.9	244.9	20
Fipronil	14.18	368.9	214.9	30
Fluazifop-P-butyl	16.91	282.1	91.1	18
Fluazifop-P-butyl	16.91	282.1	238.1	16
Fluazifop-P-butyl	16.91	383.1	282.1	14
Fluchloralin	10.76	306.0	264.0	8
Fluchloralin	10.76	264.0	206.0	8
Fluchloralin	10.76	326.0	63.0	12
Fluchloralin	10.76	264.0	159.5	14
Flucythrinate peak 1	25.65	157.0	107.1	12
Flucythrinate peak 1	25.65	199.1	107.1	22
Flucythrinate peak 1	25.65	199.1	157.1	8
Flucythrinate peak 2	26.03	157.0	107.0	12
Flucythrinate peak 2	26.03	199.0	107.0	22
Flucythrinate peak 2	26.03	199.0	157.1	8
Fludioxonil	15.82	248.0	127.0	26
Fludioxonil	15.82	248.0	154.0	18
Fludioxonil	15.82	248.0	182.0	10
Fludioxonil	15.82	153.7	127.0	8
Fluquinconazole	23.87	340.0	298.0	16
Fluquinconazole	23.87	340.0	108.1	36
Fluquinconazole	23.87	340.0	313.0	14
Fluridone	26.31	328.1	189.1	38
Fluridone	26.31	328.1	258.8	24
Fluridone	26.31	329.1	328.5	12
Flusilazole	16.32	233.1	164.9	16
Flusilazole	16.32	233.1	151.9	14
Flusilazole	16.32	315.1	233.1	10
Flusilazole	16.32	206.0	151.3	14
Flutolanil	15.73	173.0	95.0	28
Flutolanil	15.73	281.0	173.0	10
Flutolanil	15.73	173.0	145.0	14
Flutriafol	15.57	123.0	75.0	24
Flutriafol	15.57	219.1	95.0	34
Flutriafol	15.57	219.1	123.0	12
Flutriafol	15.57	123.0	95.0	12
Fluvalinate peak 1	27.25	250.0	55.1	16
Fluvalinate peak 1	27.25	250.0	199.9	18
Fluvalinate peak 1	27.25	180.8	152.1	22
Fluvalinate peak 2	27.38	250.0	55.1	16
Fluvalinate peak 2	27.38	250.0	200.0	16
Fluvalinate peak 2	27.38	180.8	152.1	20
Folpet	14.77	261.9	130.0	14
Folpet	14.77	259.9	130.0	14
Folpet	14.77	104.0	76.0	10
Folpet	14.77	130.0	102.0	12
Fonofos	10.75	137.0	109.0	6
Fonofos	10.75	109.0	62.9	10
Fonofos	10.75	246.0	109.0	14
Fonofos	10.75	246.0	137.0	6
Heptachlor	12.35	271.8	236.8	12
Heptachlor	12.35	273.8	238.8	14
Heptachlor	12.35	273.8	236.8	14
Heptachlor	12.35	99.8	39.0	26
Heptachlor	12.35	99.8	65.0	12
Heptachlor epoxide	14.39	352.8	262.9	16
Heptachlor epoxide	14.39	354.7	264.9	12
Heptachlor epoxide	14.39	262.9	192.9	30
Hexachlorobenzene	10.03	281.8	211.8	28
Hexachlorobenzene	10.03	283.8	213.8	30
Hexachlorobenzene	10.03	283.8	248.8	16
Hexachlorobenzene	10.03	285.8	250.8	18
Hexazinone	18.82	171.1	71.1	14
Hexazinone	18.82	171.1	85.1	12
Hexazinone	18.82	127.7	83.0	10

Appendix 2 (continued). Target list of analytes with their SRM parameters

Compound Name	RT (min)	Q1	Q3	CE
Iodofenfos	15.76	376.8	361.8	16
Iodofenfos	15.76	378.8	363.8	14
Iodofenfos	15.76	125.0	47.0	12
Iodofenfos	15.76	125.0	79.0	6
Iodofenfos	15.76	376.8	361.8	16
Iprodione	20.03	314.0	245.0	10
Iprodione	20.03	315.7	247.0	10
Iprodione	20.03	315.7	273.0	8
Isazophos	11.00	160.9	119.0	8
Isazophos	11.00	118.9	76.0	18
Isazophos	11.00	256.9	161.9	4
Isazophos	11.00	161.0	146.0	6
Isodrin	14.13	192.9	157.0	20
Isodrin	14.13	146.9	111.1	10
Isodrin	14.13	192.9	123.0	28
Isopropalin	13.87	280.1	238.2	8
Isopropalin	13.87	280.1	180.2	10
Isopropalin	13.87	264.1	222.1	6
Isopropalin	13.87	280.1	117.8	26
Lenacil	18.52	153.0	135.6	12
Lenacil	18.52	153.0	82.1	16
Lenacil	18.52	153.0	110.0	14
Leptophos	21.51	171.0	77.1	18
Leptophos	21.51	171.0	51.0	38
Leptophos	21.51	171.0	124.3	10
Linuron	12.88	248.0	61.1	8
Linuron	12.88	159.8	133.0	12
Linuron	12.88	187.0	124.0	20
Malathion	12.90	158.0	125.0	6
Malathion	12.90	173.1	99.0	12
Malathion	12.90	127.0	99.0	6
Malathion	12.90	92.8	63.0	8
Malathion	12.90	125.0	79.0	8
Metalaxyl	12.25	234.1	146.1	20
Metalaxyl	12.25	249.1	190.1	6
Metalaxyl	12.25	234.1	174.1	10
Metazachlor	14.13	209.0	132.1	16
Metazachlor	14.13	133.1	132.1	12
Metazachlor	14.13	132.1	117.1	14
Metazachlor	14.13	133.1	117.3	22
Methacrifos	7.96	207.9	180.1	6
Methacrifos	7.96	124.9	47.1	12
Methacrifos	7.96	125.0	79.0	8
Methacrifos	7.96	180.0	93.0	10
Methacrifos	7.96	240.0	180.0	10
Methoxychlor	20.56	227.1	141.1	32
Methoxychlor	20.56	227.1	169.1	22
Methoxychlor	20.56	227.1	212.1	12
Metolachlor	13.10	238.1	162.1	10
Metolachlor	13.10	238.1	133.1	26
Metolachlor	13.10	162.1	133.1	14
Mevinphos	7.43	127.0	109.0	10
Mevinphos	7.43	127.0	95.0	14
Mevinphos	7.43	192.0	127.0	10
MGK-264 A	13.80	164.1	98.1	10
MGK-264 A	13.80	164.1	80.1	24
MGK-264 A	13.80	164.1	93.1	10
MGK-264 B	14.17	164.1	98.1	12
MGK-264 B	14.17	164.1	67.1	6
MGK-264 B	14.17	164.1	80.1	22
Mirex	22.22	272.0	236.8	14
Mirex	22.22	273.8	238.8	14
Mirex	22.22	236.8	142.9	26
Myclobutanil	16.23	179.0	125.0	14
Myclobutanil	16.23	179.0	90.0	28
Myclobutanil	16.23	150.0	123.0	14
Myclobutanil	16.23	179.0	151.9	8
N-(2,4-Dimethylphenyl)formamide	7.90	149.1	106.1	16
N-(2,4-Dimethylphenyl)formamide	7.90	149.1	120.1	14
N-(2,4-Dimethylphenyl)formamide	7.90	149.1	121.1	6
Nitralin	19.46	316.2	274.0	8
Nitralin	19.46	274.0	216.0	8
Nitralin	19.46	274.0	169.0	12
Nitrofen	16.88	202.0	139.0	24
Nitrofen	16.88	283.0	162.0	20
Nitrofen	16.88	283.0	253.0	10
Nonachlor-cis	17.48	408.8	299.9	18
Nonachlor-cis	17.48	406.8	299.9	14
Nonachlor-cis	17.48	262.9	192.9	28
Nonachlor-cis	17.48	410.8	301.8	14
Nonachlor-cis	17.48	236.7	142.9	24
Nonachlor-trans	15.59	408.8	299.8	18
Nonachlor-trans	15.59	406.8	299.8	14
Nonachlor-trans	15.59	271.8	236.8	14
Nonachlor-trans	15.59	408.8	301.8	14
Nonachlor-trans	15.59	236.8	142.9	24

Appendix 2 (continued). Target list of analytes with their SRM parameters

Compound Name	RT (min)	Q1	Q3	CE
Nonachlor-trans	15.59	262.8	192.9	28
Norflurazon	18.36	303.0	145.0	20
Norflurazon	18.36	145.0	95.0	16
Norflurazon	18.36	145.0	74.7	28
Ortho-phenylphenol	8.24	170.1	141.1	22
Ortho-phenylphenol	8.24	141.1	115.1	14
Ortho-phenylphenol	8.24	170.1	115.0	34
Oxadiazon	16.14	175.0	112.0	12
Oxadiazon	16.14	175.0	76.0	28
Oxadiazon	16.14	258.0	175.0	6
Oxadiazon	16.14	174.9	147.2	6
Oxyfluorfen	16.30	300.0	223.0	14
Oxyfluorfen	16.30	252.0	146.0	30
Oxyfluorfen	16.30	252.0	169.8	28
Paclobutrazol	15.22	236.0	125.0	12
Paclobutrazol	15.22	236.0	167.0	10
Paclobutrazol	15.22	138.0	103.1	14
Paclobutrazol	15.22	125.0	89.0	18
Parathion (ethyl)	13.33	291.0	109.0	12
Parathion (ethyl)	13.33	138.9	109.0	6
Parathion (ethyl)	13.33	154.9	125.0	6
Parathion (ethyl)	13.33	109.0	81.0	10
Leptophos	21.51	171.0	77.1	18
Leptophos	21.51	171.0	51.0	38
Leptophos	21.51	171.0	124.3	10
Linuron	12.88	248.0	61.1	8
Linuron	12.88	159.8	133.0	12
Linuron	12.88	187.0	124.0	20
Parathion (ethyl)	13.33	124.9	97.0	6
Parathion-methyl	12.04	263.0	109.0	12
Parathion-methyl	12.04	233.0	109.0	10
Parathion-methyl	12.04	263.0	79.0	30
Parathion-methyl	12.04	124.9	47.0	12
Parathion-methyl	12.04	124.9	79.0	6
Pebulate	7.71	128.1	57.1	8
Pebulate	7.71	128.0	72.0	6
Pebulate	7.71	161.0	128.1	6
Pebulate	7.71	108.1	77.1	24
Pebulate	7.71	108.1	79.0	14
Penconazole	14.26	248.1	157.0	22
Penconazole	14.26	248.1	192.0	12
Penconazole	14.26	159.0	123.0	20
Penconazole	14.26	158.9	89.0	28
Pendimethalin	14.04	252.1	162.1	8
Pendimethalin	14.04	252.1	161.1	14
Pendimethalin	14.04	252.1	191.3	8
Pentachloroaniline	11.64	262.9	191.9	20
Pentachloroaniline	11.64	264.9	193.6	18
Pentachloroaniline	11.64	266.9	193.9	20
Pentachloroaniline	11.64	264.8	202.8	20
Pentachloroaniline	11.64	264.8	229.3	12
Pentachloroanisole	10.12	264.8	236.9	10
Pentachloroanisole	10.12	266.8	238.9	10
Pentachloroanisole	10.12	279.9	236.8	22
Pentachlorobenzene	8.28	249.8	214.8	16
Pentachlorobenzene	8.28	247.9	212.9	18
Pentachlorobenzene	8.28	248.0	142.0	42
Pentachlorobenzene	8.28	249.8	143.6	38
Pentachlorobenzene	8.28	249.8	178.5	24
Pentachlorobenzonitrile	10.60	272.9	237.9	16
Pentachlorobenzonitrile	10.60	274.8	204.9	28
Pentachlorobenzonitrile	10.60	274.8	239.9	18
Pentachlorothioanisole	12.91	295.7	262.9	12
Pentachlorothioanisole	12.91	262.7	192.9	28
Pentachlorothioanisole	12.91	295.7	245.9	30
Permethrin peak 1	23.65	183.1	153.0	12
Permethrin peak 1	23.65	183.1	168.0	12
Permethrin peak 1	23.65	163.0	91.1	12
Permethrin peak 2	23.91	183.0	165.1	10
Permethrin peak 2	23.91	183.0	153.0	14
Permethrin peak 2	23.91	183.0	168.1	10
Perthane (Ethylan)	16.93	223.1	167.0	12
Perthane (Ethylan)	16.93	223.1	179.0	20
Perthane (Ethylan)	16.93	223.1	193.0	28
Phenothrin	21.38	123.1	81.1	8
Phenothrin	21.38	123.1	41.1	24
Phenothrin	21.38	123.1	79.1	14
Phorate	9.75	260.0	75.0	8
Phorate	9.75	121.0	65.0	10
Phorate	9.75	75.0	47.0	8
Phosalone	21.47	182.0	74.8	30
Phosalone	21.47	121.1	65.0	10
Phosalone	21.47	182.0	111.0	14
Phosmet	20.17	160.0	76.9	22
Phosmet	20.17	160.0	133.0	10
Phosmet	20.17	160.0	50.9	38
Phthalimide	7.80	147.0	76.0	25
Phthalimide	7.80	147.0	103.3	10
Phthalimide	7.80	103.7	76.0	10
Piperonyl butoxide	19.46	176.1	103.1	22
Piperonyl butoxide	19.46	176.1	117.0	18
Piperonyl butoxide	19.46	176.1	131.1	12
Pirimiphos-ethyl	13.72	304.1	168.1	12
Pirimiphos-ethyl	13.72	318.1	166.1	12
Pirimiphos-ethyl	13.72	318.1	182.1	10
Pirimiphos-methyl	12.61	233.0	151.1	6
Pirimiphos-methyl	12.61	290.1	125.0	20
Pirimiphos-methyl	12.61	290.1	233.0	8

Appendix 2 (continued). Target list of analytes with their SRM parameters

Compound Name	RT (min)	Q1	Q3	CE
Pirimiphos-methyl	12.61	305.1	180.1	8
Pretilachlor	15.94	162.0	132.1	20
Pretilachlor	15.94	262.1	202.1	6
Pretilachlor	15.94	176.1	147.1	14
Pretilachlor	15.94	202.1	174.2	8
Pretilachlor	15.94	202.1	145.5	14
Pretilachlor	15.94	238.1	146.1	10
Prochloraz	24.01	308.0	70.0	12
Prochloraz	24.01	180.0	69.0	14
Prochloraz	24.01	180.1	138.1	12
Prochloraz	24.01	69.9	42.0	8
Prochloraz	24.01	308.0	147.1	12
Procymedone	14.66	283.0	96.1	8
Procymedone	14.66	283.0	68.1	24
Procymedone	14.66	285.0	96.1	10
Procymedone	14.66	95.9	53.0	16
Procymedone	14.66	95.9	67.1	8
Prodiamine	12.64	321.1	279.1	6
Prodiamine	12.64	275.1	255.1	8
Prodiamine	12.64	321.1	203.0	10
Prodiamine	12.64	279.0	203.1	6
Profenofos	16.00	338.9	268.9	14
Profenofos	16.00	336.9	266.9	12
Profenofos	16.00	296.9	268.9	10
Profenofos	16.00	336.9	308.9	8
Profluralin	10.49	318.1	199.1	12
Profluralin	10.49	318.1	55.0	12
Profluralin	10.49	347.1	330.1	6
Profluralin	10.49	318.1	284.1	10
Profluralin	10.49	330.2	69.1	20
Propachlor	8.94	120.0	50.9	32
Propachlor	8.94	120.0	77.0	16
Propachlor	8.94	176.1	57.1	8
Propanil	11.79	217.0	161.0	8
Propanil	11.79	161.0	90.0	24
Propanil	11.79	161.0	99.0	24
Propanil	11.79	160.9	125.7	16
Propargite	19.26	135.1	107.1	12
Propargite	19.26	135.1	77.1	26
Propargite	19.26	150.1	135.1	8
Propisochlor	12.20	162.1	144.1	8
Propisochlor	12.20	162.1	91.1	30
Propisochlor	12.20	162.1	120.1	12
Propisochlor	12.20	162.1	147.1	12
Propyzamide	10.70	172.9	109.0	24
Propyzamide	10.70	172.9	145.0	14
Propyzamide	10.70	174.9	147.0	14
Propyzamide	10.70	172.9	74.0	38
Prothiofos	15.87	266.9	220.9	18
Prothiofos	15.87	266.9	238.9	8
Pentachlorothioanisole	12.91	295.7	262.9	12
Pentachlorothioanisole	12.91	262.7	192.9	28
Pentachlorothioanisole	12.91	295.7	245.9	30
Permethrin peak 1	23.65	183.1	153.0	12
Permethrin peak 1	23.65	183.1	168.0	12
Permethrin peak 1	23.65	163.0	91.1	12
Prothiofos	15.87	309.0	238.9	14
Pyraclofos	23.00	194.0	138.0	18
Pyraclofos	23.00	360.0	194.1	12
Pyraclofos	23.00	139.2	96.9	6
Pyrazophos	22.48	221.0	193.1	8
Pyrazophos	22.48	231.9	204.1	10
Pyrazophos	22.48	221.0	148.7	14
Pyridaben	23.90	147.1	117.1	20
Pyridaben	23.90	147.1	132.1	12
Pyridaben	23.90	147.1	119.1	8
Pyridaphenthion	19.97	340.0	199.1	8
Pyridaphenthion	19.97	199.0	77.1	24
Pyridaphenthion	19.97	199.0	92.1	14
Pyrimethanil	10.89	198.1	118.0	32
Pyrimethanil	10.89	198.1	158.1	18
Pyrimethanil	10.89	198.1	183.1	14
Pyriproxyfen	21.81	136.1	78.0	20
Pyriproxyfen	21.81	136.1	96.0	10
Pyriproxyfen	21.81	226.1	186.1	12
Quinalphos	14.55	146.0	118.1	10
Quinalphos	14.55	157.1	102.0	22
Quinalphos	14.55	157.1	129.0	14
Quintozene	10.53	294.8	236.9	14
Quintozene	10.53	213.8	178.9	14
Quintozene	10.53	213.8	141.9	28
Resmethrin peak 1	19.34	171.0	127.9	14
Resmethrin peak 1	19.34	143.0	128.1	10
Resmethrin peak 1	19.34	123.1	81.1	8
Resmethrin peak 2	19.54	171.0	127.9	14
Resmethrin peak 2	19.54	143.0	128.0	10
Resmethrin peak 2	19.54	123.1	81.1	8
Sulfotep	9.48	202.0	145.9	10
Sulfotep	9.48	237.9	145.9	12
Sulfotep	9.48	322.0	145.9	22
Sulfotep	9.48	265.9	145.9	15
Sulprofos	17.97	322.0	156.1	10
Sulprofos	17.97	156.0	108.0	30
Sulprofos	17.97	156.0	141.0	14
Tebuconazole	19.13	250.0	125.0	20
Tebuconazole	19.13	125.0	89.0	16
Tebuconazole	19.13	125.0	99.0	16
Tebufenpyrad	20.85	276.1	171.0	10

Appendix 2 (continued). Target list of analytes with their SRM parameters

Compound Name	RT (min)	Q1	Q3	CE
Tebufenpyrad	20.85	318.1	131.1	14
Tebufenpyrad	20.85	318.1	145.1	14
Tecnazene	8.85	258.9	201.0	12
Tecnazene	8.85	202.9	142.9	18
Tecnazene	8.85	214.8	178.9	8
Tecnazene	8.85	214.8	143.6	20
Tecnazene	8.85	214.8	179.9	15
Tefluthrin	11.01	177.0	127.0	14
Tefluthrin	11.01	177.0	137.0	16
Tefluthrin	11.01	197.0	141.0	10
Terbacil	11.00	161.0	144.0	12
Terbacil	11.00	160.0	117.0	8
Terbacil	11.00	160.0	76.0	12
Terbufos	10.63	231.0	128.9	20
Terbufos	10.63	231.0	175.0	10
Terbufos	10.63	231.0	203.0	8
Terbuthylazine	10.62	214.1	104.1	16
Terbuthylazine	10.62	214.1	132.1	10
Terbuthylazine	10.62	229.1	173.1	10
Tetrachlorvinphos	15.17	328.9	109.0	18
Tetrachlorvinphos	15.17	330.9	109.0	18
Tetrachlorvinphos	15.17	332.9	109.0	14
Tetrachlorvinphos	15.17	109.0	79.0	6
Tetradifon	21.23	159.0	131.0	10
Tetrahydrophthalimide (THPI)	7.97	151.0	79.9	6
Tetrahydrophthalimide (THPI)	7.97	151.0	77.1	32
Tetrahydrophthalimide (THPI)	7.97	151.0	122.1	8
Tetramethrin peak 1	20.12	164.0	107.1	12
Tetramethrin peak 1	20.12	164.0	77.1	24
Tetramethrin peak 1	20.12	164.0	135.1	8
Tetramethrin peak 2	20.43	164.0	107.1	12
Tetramethrin peak 2	20.43	164.0	77.1	22
Tetramethrin peak 2	20.43	164.0	135.1	8
Tolclofos-methyl	12.09	265.0	250.0	12
Tolclofos-methyl	12.09	265.0	219.9	20

Compound Name	RT (min)	Q1	Q3	CE
Tolclofos-methyl	12.09	266.9	252.0	12
Tolylfluanid	14.34	238.0	137.0	10
Tolylfluanid	14.34	137.0	65.1	28
Tolylfluanid	14.34	137.0	91.1	18
Tolylfluanid	14.34	238.0	91.0	40
Tolylfluanid	14.34	240.0	137.0	14
Transfluthrin	12.10	163.0	143.0	14
Transfluthrin	12.10	127.0	91.1	8
Transfluthrin	12.10	163.0	91.1	12
Triadimefon	13.43	208.0	111.0	20
Triadimefon	13.43	208.0	126.7	12
Triadimefon	13.43	208.0	180.8	8
Triadimenol	14.66	168.1	70.0	10
Triadimenol	14.66	128.0	65.0	18
Triadimenol	14.66	112.0	58.0	8
Triadimenol	14.66	128.0	100.0	10
Triallate	11.21	268.0	183.9	18
Triallate	11.21	86.1	43.3	6
Triallate	11.21	268.0	226.0	12
Triazophos	17.92	161.1	134.1	8
Triazophos	17.92	257.0	162.1	6
Triazophos	17.92	161.1	106.1	12
Triazophos	17.92	162.1	119.1	12
Triflumizole	14.72	206.0	179.0	14
Triflumizole	14.72	179.0	144.0	14
Triflumizole	14.72	206.0	186.0	8
Trifluralin	9.37	306.1	264.1	8
Trifluralin	9.37	264.0	160.0	14
Trifluralin	9.37	306.1	206.0	10
Triphenylphosphate	19.28	215.0	168.1	16
Triphenylphosphate	19.28	326.1	325.1	10
Triphenylphosphate	19.28	326.1	169.1	28
Vinclozolin	11.98	186.8	124.0	18
Vinclozolin	11.98	197.9	145.0	14
Vinclozolin	11.98	212.0	172.0	14

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